DEPARTMENT OF AGRICULTURE.

DIVISION OF CHEMISTRY.

BULLETIN No. 6.

EXPERIMENTS

WITH

DIFFUSION AND CARBONATATION

AT

OTTAWA, KANSAS,

CAMPAIGN OF 1885,

BY

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EXPERIMENTS IN THE MANUFACTURE OF SUGAR AT OTTAWA, KANS., SEASON OF 1885.

OTTAWA, KANS., *October* 16, 1885.

Hon. NORMAN J. COLMAN,

Commissioner :

SIR : I submit herewith a report of the experiments made by the Department of Agriculture in the manufacture of sugar from sorghum at Ottawa, Kans., during the season of 1885.

It may be of interest to you to know what had been done in the matter before you took office. The appropriation bill for the Department of Agriculture, for the fiscal year beginning July 1, 1884, was approved June 5, 1884. The bill contained an item of $50,000 for "necessary expenses in conducting experiments, including experiments in the manufacture of sugar from sorghum and other vegetable plants." About the end of June, 1884, the Commissioner of Agriculture informed me that he had determined to try the process of diffusion on sorghum cane in Kansas during the season of 1884. At that time (the appropriations could not be used until July 1) I represented to the Commissioner that it would be impossible to build and erect an apparatus by September 1, the time when the manufacturing season would open. I suggested that the whole subject be carefully investigated, trials made with the different forms of cane-slicers and apparatus prepared for the season of 1885. This view did not meet with the approval of the Commissioner, who expressed a desire to comply as speedily as possible with the wish of Congress to have the experiments made. Accordingly, at his request, representatives of two manufacturing firms, viz, The Colwell Iron Company of New York, and the Pusey & Jones Company of Wilmington, Del., came to Washington and conferred with him in respect of the matter. The Colwell Company refused to undertake the work at all, and the Pusey & Jones Company agreed to try it and deliver the apparatus by September 15, provided the order was given prior to July 10. In spite of the fact that the company did not undertake to finish the battery until after the manufacturing season would have begun, and that two or three months at least would have been required for the shipment and erection of the apparatus, the order was given July 14, 1884, to go ahead with the erection of the apparatus. The Commissioner requested me to aid the contractors as much as pos-

sible in securing an outfit suitable for the purpose, but no time was afforded to elaborate plans since work had to be commenced at once in order that the apparatus be ready by the time specified. I had little hope of success with apparatus constructed in such a way, but when near the end of August it became evident that it could not be finished what little I had vanished. An attempt was then made to have the apparatus ready for use in Louisiana during the course of 1884, but this effort had also to be abandoned by reason of delay in finishing the work.

Late in the fall of 1884 the apparatus was finished and ready for inspection. I had requested that the cells of the battery be large enough to contain one ton of cane chips, and the builders furnished for this capacity a cell measuring 3 feet bottom diameter, 3 feet 11 inches shoulder diameter, 1 foot 2½ inches depth of neck and 16 inches diameter of neck, 1 foot shoulder to bottom of neck, and 6 feet deep, containing 66 (nearly) cubic feet of space.

In the absence of any experience which would enable me to judge definitely of the matter, it appeared that the cells were sufficiently large, and they were therefore accepted. The cutters, however, appeared to be entirely inadequate to the work to be performed, and they were therefore rejected.

No further action was taken by the Commissioner in the matter except to request the company to store the apparatus during the winter until February 13, 1885, when the Commissioner sent for me and asked me to go to New York and make a contract with Mr. B. Urner, president of the Franklin Sugar Company at Ottawa, Kans., for the erection of the battery in connection with the works of his company at Ottawa. I met Mr. Urner at the Astor House on the 13th of February, and we agreed upon the form of a contract which I made out in duplicate to be signed by Mr. Urner and the Commissioner.

In March, 1885, the cells were sent out to Ottawa, but no action was taken in respect of the cutters.

This was the condition of affairs at the time you took office.

Following is a record of the work which has been done by me under your supervision. It was discovered, on consulting the books of the disbursing officer on May 1, 1885, that only a little over $1,000 of the appropriation remained available. The new appropriation of $40,000 for 1885–'86 could not be used until July 1. On May 8 you appointed M. A. Scovell to superintend the erection of the machinery and buildings at Ottawa.

He was instructed to do what he could with the small amount of funds available.

In the middle of June I went to Ottawa to assist Mr. Scovell in locating the buildings and getting the work under way.

On the 3d of July, the new appropriation having become available, I went to Wilmington, Del., and arranged for the construction of the new cutters. A few days later you ordered of the same firm the neces-

sary machinery to operate the cutters and convey the chips to the diffusion cells.

The work of construction was hurried as rapidly as possible, and the first consignment of apparatus, consisting of one cutter and shafting, pulleys, hangers, belting, &c., reached Ottawa on Saturday, September 4, ten days after the manufacturing season had commenced.

I reached Ottawa on Wednesday, September 8, and the work of erection was pushed with all possible dispatch. On Sunday, September 27, the rest of the machinery arrived. During the following week, one cutter having been completed, preliminary trials were made with the apparatus. The cutter was found to give good satisfaction, with a capacity of 6 tons per hour, giving a nicely grooved chip well suited for diffusion.

On the other hand, the cells of the battery were found to hold only 1,340 pounds (or, by a little crowding, 1,400 pounds), instead of a ton; the lower opening, through which the chips were to be discharged, was too small, not allowing the chips to flow out freely, and requiring the services of two additional laborers to empty the cell. The conveyor, bringing the chips to the cells, would not work automatically, as had been intended, and a third man was required to direct the chips into the cells. Owing to these faults of construction, which could not have been foreseen, the expense of working the battery was almost double what it would be with proper fixtures. Add to this the additional expense occasioned by the small capacity of the cells and a serious increase of working expenses is at once apparent.

The battery was intended by the constructors to be worked by allowing the liquid to enter from the bottom of each cell, forcing the heavier liquid out at the top. Numerous trials by this method showed that it was impracticable. The liquid entering the cell is both lighter and warmer than that contained in it. The result of the lighter liquid being below was an admixture of the contained and entering liquors, which proved disastrous to close extraction. As a consequence the connection with the water service had to be changed so as to permit downward working.

On Sunday morning, October 4, a heavy frost killed the blades of the cane, but did no damage to that which was fully matured. On Tuesday morning, October 6, there was a severe freeze, ice having formed an eighth of an inch in thickness. This freeze did a great injury to the cane, as will be seen in the table of analysis of the mill juices. On Wednesday, October 7, the machinery of the diffusion battery, although hastily and imperfectly put together, was pronounced ready for trial.

On Thursday, October 8, our first trial took place. The cutters were started at 8 a. m., and work was continued until 5 a. m. Friday, October 9. During this time 70 cells were diffused of 1,400 pounds each, or

a total of 98,000 pounds. The weight of the diffused juice was 96,140 pounds from 65 cells.

The analysis of the exhausted chips showed only a trace of sucrose and 0.10 per cent. of glucose. The waste waters of diffusion showed a loss of 0.10 per cent. sucrose and 0.10 per cent. glucose. The total loss of sugars, therefore, was 0.10 per cent. of sucrose and 0.20 per cent. glucose. The excess of glucose in these analyses is explained by the fact that the samples of exhausted chips and waste waters taken during the night were not analyzed until the next day, and meanwhile the sucrose suffered inversion.

The total loss of sugar, therefore, in chips and waste waters was .30 per cent. This is a remarkably good extraction, and the result is very satisfactory.

When the cells could be promptly emptied it was an easy matter to make a diffusion every twelve minutes, and the extraction was just as good as in those cases where twice that time was employed. These results showed that sorghum cane diffuses with great readiness, and in this respect it appears to have an advantage over the beet.

The cane employed was quite imperfectly stripped and the sheaths and remaining blades were, of course, treated in the cells together with the chips. The coloring and gummy matters which they contained were, therefore, found in the diffusion juices.

It will be a very easy matter to run these chips through a sieve in connection with a blower and remove all objectionable matter.

Following is a table showing the details of the diffusion.

Table of diffusion.

Number of cell.	Volume of juice drawn off.	Temperature of juice.	Temperature first preceding cell.	Temperature second preceding cell.	Specific gravity.	Time.
	Liters.	°C.	°C.	°C.		
1	700	22	60	77	1.050	9.35 a. m.
2	700	19	61	75	1.031	9.44
3	700	24	72	64	1.038	9.47
4	700	20	60	64	1.040	10.13
5	700	19	62	59	1.041	10.25
6	700	24	....	58	1.045	10.27
7	700	19	61	....	1.045	10.39
8	700	25	60	58	1.042	10.51
9	700	27	58	60	1.042	11.20
10	700	27	59	58	1.041	11.37
11	700	29	64	56	1.041	11.50
12	700	24	....	67	1.039	12.02 p. m.
13	700	24	60	52	1.039	12.14
		Stopped for dinner.				
14	700	27	50	56	1.026	1.25
15	700	25	....	50	1.036	1.35
16	700	23	50	....	1.037	1.47
17	700	23	54	50	1.038	1.58
18	700	25	49	55	1.037	2.10
19	700	27	52	53	1.037	2.22
20	700	25	....	....	1.035	2.34
21	700	22	58	....	1.038	2.50
22	700	24	60	....	1.037	3.02
23	700	24	66	54	1.037	3.14
24	700	25	67	59	1.038	3.26

Table of diffusion—Continued.

Number of cell.	Volume of juice drawn off.	Temperature of juice.	Temperature first preceding cell.	Temperature second preceding cell.	Specific gravity.	Time.
	Liters.	° C.	° C.	° C.		
25	700	25	....	56	1.039	3.41 p. m.
26	700	26	64	....	1.040	3.53
27	700	23	64	55	1.039	4.06
28	700	23	66	55	1.040	4.23
29	700	26	65	54	1.039	4.36
30	700	25	58	50	1.036	4.52
31	700	24	56	50	1.039	5.15
32	....	....	64	56	1.039	5.27
33	600	26	38	59	1.039	5.42
		Stopped for supper.				
34	600	22	....	....	1.034	7.15
35	600	....	38	59	....	7.30
36	600	19	46	58	1.036	7.50
37	600	18	54	58	1.038	8.12
38	600	22	54	58	1.034	8.37
39	600	19	56	64	1.040	8.50
40	600	28	62	....	1.040	9.00
41	600	21	65	56	1.041	10.05
42	600	24	65	56	1.042	10.19
43	600	23	....	56	1.043	10.31
44	600	23	65	....	1.042	10.47
45	600	27	64	54	1.041	11.09
46	600	30	68	56	1.040	11.20
47	600	26	68	56	1.040	11.37
48	600	24	68	56	1.040	11.59
49	600	23	64	58	1.042	12.48 a. m.
50	600	24	68	....	1.042	1.14
51	600	22	68	56	1.041	1.26
52	600	23	63	54	1.040	1.55
53	600	27	....	58	1.039	2.17
54	600	24	66	....	1.041	2.31
55	600	24	65	57	1.042	3.02
56	600	27	68	58	1.041	3.15
57	600	25	68	57	1.041	3.27
58	600	22	59	54	1.036	3.45
59	600	21	58	54	1.041	3.59
60	600	23	....	55	1.042	4.13
61	600	61	67	62	1.042	4.28
62	600	20	67	62	1.042	4.40
63	600	19	....	43	1.053	4.53

Two more portions of 600 liters each were drawn off and the other five cells were then discarded.

In the first series of 32 cells 700 liters of juice were drawn off at each charge, or 1,540 pounds. Since the average charge of each cell was 1,400 pounds of chips the ratio of diffusion juice to weight of cane was 110:100. In the second series of 31 cells 600 liters of juice were drawn off at each charge, or 1,320 pounds, the ratio of diffusion juice to weight of cane was 94.3:100. It appears from this that diffusion can be successfully practiced with sorghum cane when the weight of juice obtained is made about the same as that of the cane diffused. The mean specific gravity of the 32 charges of 700 liters each was 1.0394 at 25° or at 15° 1.0411, corresponding to 10.24 per cent. total solids. The average of specific gravity of the juice of 32 charges of 600 liters each was 1.0405 at 25°, or 1.0424, corresponding to 10.55 per cent. total solids. The cane varied so greatly in its composition that no estimate of the degree of extraction could be made from the analyses of the cane juices.

The following analyses were made of the diffusion juices during the day:

	First time 10.30 a. m.	Second time 3 p. m.
	Per cent.	*Per cent.*
Total solids	10.84	9.70
Glucose	2.32	2.00
Sucrose	6.19	5.90
Solids not sugar	2.33	1.80

No analyses of the diffusion juice were made at night.

Analyses were also made of the cane juice expressed on small mill from canes taken from the yard whence the cutter carriers were supplied.

	First analysis, 10 a. m.	Second analysis, 11 a. m.	Third analysis, 11.30 a. m.
	Per cent.	*Per cent.*	*Per cent.*
Total solids	17.00	15.60	15.20
Sucrose	11.24	9.62	9.83
Glucose	2.44	2.85	3.41
Not sugar	3.32	3.13	1.96

The small samples taken for the above analyses showed how seriously the frost had affected the canes and how irregular they were in their composition. The peculiar odor characteristic of frosted sorghum was also very apparent in the chips as they came from the cutter. This condition of things rendered this part of the work very unsatisfactory.

The weight of coal used from the time of firing up on Thursday morning until the time of stopping Friday morning—nearly 24 hours—was a little over 3,000 pounds. But when the chips could be promptly removed from the cells it was possible to make a diffusion every 10 minutes. Hence the whole work might have been done in 700 minutes, or 11 hours and 40 minutes. In this case half the coal might have been saved.

The force required to do the work was:

1 fireman (day) and 1 (night), at $1.50	$3 00
4 men on cane-carrier (day) and 4 (night), at $1.25	10 00
4 men at battery (day) and 4 (night), at $1.25	10 00
1 team to remove chips (day) and 1 (night), at $2.50	5 00
1 valve-man (day) and 1 (night), at $2.25	4 50
1½ tons coal, at $3.25	4 88
Oil and lights	1 00
1 boy (to sweep, &c.)	75
Total cost of diffusing 49 tons of cane	38 13

With slight changes in the battery it will be an easy matter to reduce this to $20, and if the cells are made twice as large the cost will be still less. With apparatus properly arranged the cost of diffusing a ton of cane will not be greater than 30 cents. Good machinists estimated that

about 15 horse-power was used in driving the machinery and heating the cells in the experiments made.

A study of the table of diffusions reveals some curious facts relating to the variations in the composition of the cane. Beginning with the fourth diffusion and ending with the eleventh, eight cells in all, the following facts appear: The mean specific gravity of the diffusion juice at 15° was 1.045, corresponding to 11.2 per cent. total solids. The weight of juice drawn off was 10 per cent. greater than the weight of cane diffused; 11.2 total solids in 110 pounds would be equal to 12.32 total solids in 100 pounds. Add to this the .30 sugar lost in chips in water of diffusion and .15 not sugar, we obtain 12.77 as the total solids in cane diffused calculated from the diffusion data. This would correspond to 14.05 total solids in the juice as expressed by mill.

After the eleventh diffusion the cane rapidly deteriorated. The mean specific gravity from the eleventh to the thirty-third diffusion was 10.39 (*circa*) at 15°, corresponding to 9.75 per cent. total solids. Proceeding as above, the following numbers are obtained: Total solids in cane dif. fused, 11.18 per cent. This would give for mill juice 12.35 per cent.

After the thirty-second diffusion the weight of juice drawn off was 5 per cent. less than weight of cane diffused. The specific gravity of the diffusion juice at 15° was 1.044 (*circa*), corresponding to 11 per cent. total solids. Diminishing this by 5 per cent. and adding .45 we obtain 11 as total solids in cane or 12.10 per cent. total solids in expressed juice.

Unfortunately the carbonatation pump broke after about one-third of the juice had been defecated. A careful estimate of the number of tons of the juice which was worked showed that 15 had been carbonated.

This yielded 4,320 pounds of *masse cuite*, containing 76.9 per cent. solid matter, or 11 per cent. (nearly) on weight of cane worked.

The composition of this *masse cuite* is shown by the following analysis:

	Per cent.
Sucrose	53.48
Glucose	13.55
Water	23.10
Ash	4.74
Not sugar	5.13

This *masse cuite* was allowed to stand in cars (it was not boiled to grain) one week, and was then swung out, yielding 1,420 pounds, or about 30 per cent. of washed and dried sugar, or 95 pounds per ton of cane worked.

Allowing 12 pounds per gallon for the *masse cuite*, the number of gallons per ton of cane was 24.0.

The sugar was of fine quality, the molasses of much better quality than that obtained in the usual way, and the whole product was in every respect satisfactory.

The cane continued to deteriorate, as is shown in the following analyses of the juices from the mill of the Franklin Sugar Company:

	Friday, October 9, 4 p. m.	Saturday, October 10, 10.30 a. m.	Saturday, October 10, 4 p. m.	Monday, October 12.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
Sucrose	7. 80	7. 32	7. 69	6. 67
Glucose	3. 50	3. 50	3. 15	3. 27
Not sugars	2. 21	3. 19	3. 50	3. 56
Total solids	13. 51	14. 01	14. 34	13. 50

These discouraging effects of the severe frost of the 6th of October did not afford any hope of a successful continuation of the experiments. It would be quite impossible to work successfully a diffusion juice derived from so poor a source.

On October 12 an analysis of some samples of cane from a different source showed that the juice had been little injured by the frost. The numbers obtained were the following:

	October 12.
	Per cent.
Sucrose	11. 83
Glucose	1. 87
Not sugars	2. 44
Total solids	16. 14

Hoping that enough of such cane might be secured to allow of another trial of the apparatus, it was determined to make another run on the following day. The sample for analysis was taken from the west end of a field which was bordered on the west by a strip of forest and a pond of water of considerable size.

Evidently the spot whence the sample was taken had been protected by these conditions from the action of the frost. Samples taken from other parts of the field during the day, October 13, when the second run was made, show, with the exception of the first one taken from the west end of the field, a serious deterioration of the cane. Following are the analyses of juices of cane expressed by experimental mill during the day of October 13.

No.	Hour.	Sucrose.	Glucose.	Not sugar.	Total solids.
1	10 a. m.	10. 23	2. 11	2. 82	15. 16
2	3 p. m.	8. 64	2. 95	2. 81	14. 40
3	4. 30 p. m.	8. 54	3. 11	2. 89	14. 54
4	5. 30 p. m.	8. 81	2. 61	2. 98	14. 40

Not only were the analyses sufficient to show the injury that had been done, but in addition to this the chips were soft and "bleeding" and possessed the odor characteristic of frozen cane.

Work with such chips was more like maceration than diffusion; nevertheless the run was made.

Run of October 13, 1885.

The cutters were started at 9.30 a. m.; stopped at 6.30 p. m.

Number of hours, nine. less 30 minutes stopped at noon, 8 hours 30 minutes.

Number of cells of chips cut, 39; average time for each cell 14 minutes (nearly).

During this time the cutters were frequently stopped to wait for the emptying of the cells.

It was found that one cutter, with moderate feed, could fill a cell (1,400 pounds *circa*) in 6 minutes.

The number of charges drawn off was 34.

The first charge was taken at 11.07 a. m.; and the last one at 7.45 p. m., less 30 minutes at noon and 30 minutes for supper, 7 hours 39 minutes.

Average time for each diffusion, 13½ minutes.

Cost.

Cost of coal, at $3.25 per ton	$2 50
Four men on carrier, eight-tenths day, at $1.25	4 00
One fireman, eight-tenths day, at $1.50	1 20
One boy, eight-tenths day, at $0.75	60
One team for bagasse, eight-tenths day, at $2.50	2 00
Four men at battery, eight-tenths day, at $1.25	4 00
One valve-man, eight-tenths day, at $2.25	2 00
Oil	50
Total	16 80

Pounds of coal burned, 1,575.

Tons cane diffused, 27.

Cost per ton, $0.62.

Table of details of diffusion of October 13, 1885.

Number of cell.	Volume of juice.	Temperature of juice.	Temperature first preceding cell.	Temperature second preceding cell.	Specific gravity.	Time.
	Liters.	°C.	°C.	°C.		
1	900	16	34	50	1.024	11.07 a. m.
2	700	18	....	50	1.037	11.23
3	700	19	48	75	1.031	11.35
4	700	18	60	62	1.033	11.48
5	700	28	56	73	1.030	12 noon.
6	700	26	....	70	1.031	12.12 p. m.
7	700	25	55	60	1.033	12.24
8	700	27	55	60	1.033	12.34
9	700	23	60	61	1.033	12.59
10	700	24	70	62	1.035	1.40
11	700	24	58	59	1.030	1.54
12	700	19	58	....	1.036	2.06
13	700	21	....	67	1.037	2.18
14	700	28	64	55	1.038	3.05
15	700	26	64	56	1.038	3.17
16	700	24	56	....	1.039	3.27
17	700	25	....	58	1.038	3.39
18	700	26	56	57	1.036	3.55
19	700	26	56	54	1.034	4.12
20	700	25	58	55	1.035	4.24
21	700	28	54	52	1.033	4.34
22	700	23	....	54	1.034	4.52
23	640	23	58	....	1.034	5.02
24	640	24	56	54	1.034	5.15
25	640	22	56	54	1.033	5.28
26	640	22	....	54	1.033	5.40
27	640	23	56	54	1.033	6.00
28	640	22	58	55	1.033	6.15
29	640	22	56	53	1.033	6.30
30	640	22	55	54	1.033	6.43
31	640	26	52	50	1.034	7.00
32	640	27	54	....	1.030	7.15
33	700	41	52	50	1.024	7.25
34	640	45	50	51	1.021	7.45

The total weight of juice delivered to liming tanks was 24,110.7 kilos., or 52,043.5 pounds.

Weight of cane in the 34 diffusion cells was 1,400 x 34=47,600 pounds. Therefore the weight of juice drawn off to cane removed was 109 to 100.

Average composition of the diffusion juices will be shown by the following analyses:

Diffusion juices.

Number.	No. of diffusion.	Sucrose.	Glucose.	Not sugar.	Total sugar.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1	4	4.86	1.69	1.78	8.33
2	14	5.94	2.00	2.20	10.14
3	21	4.99	2.31	1.64	8.94
4	25	4.76	2.25	1.55	8.56
5	31	3.91	2.16	1.63	7.70
Mean	....	4.89	2.08	1.76	8.74

Mean purity coefficient, 56.

Percentage sucrose left in exhausted chips and waste waters.

CHIPS.

Number.	No. of diffusion.	Sucrose.	Glucose.	Total sugar.
1	4	*0.06	0.062	0.122
2	14	0.049	0.031	0.080
3	31	0.068	0.083	0.148
4	25	(†)	0.125	0.125
Mean	....	....	....	0.119

* In the first analysis above the sucrose was sought for by a polariscope, but so little was present that no rotation could be observed. It is fair to presume, from a study of the analyses which follow, that 0.06 per cent. sucrose was present.

† In this analysis the chips were left over night, and in the morning no sucrose could be found. It had all been inverted, and appears, therefore, with the glucose.

WASTE WATERS.

The weight of water discharged with each cell was nearly one-third greater than that of the chips; therefore the percentages found are increased by one-third to represent the real loss.

No.	Number of diffusion.	Sucrose.	Glucose.	Total sugars.	Total sugars increased.
1	Fourth	Trace.	Trace.	....	....
2	Fourteenth	Trace.	Trace.	....	....
3	Twenty-first	0.003	0.019	0.022	0.029
4	Twenty-fifth	Trace.	0.028	0.028	0.032
	Mean	....	....	....	0.0305
	Mean total loss of sugars in chips and waste waters				0.1221

The exhaustion of the cane, therefore, is practically perfect and much superior to all expectation. With unfrozen cane I do not think it would be so perfect.

After October 13 the character of the cane made any further experiments useless, and the work for this season was therefore discontinued. The juice from the second run was all carbonated, but the process was so slow and the juice had to stand so long in contact with the lime that the product was of a much darker color than the first. The total weight of *masse cuite* obtained, 89 per cent. solid matter, was 5,510 pounds. For the twenty-three tons of cane carbonated this gives a yield of 12 per cent. (*circa*). Allowing 12 pounds per gallon, this is a yield of 240 pounds *masse cuite*, or 20 gallons per ton.

Much loss, however, was occasioned by the frequent transfer of the juice in order to secure its entire carbonatation and at the same time keep it out of the way of the other products in the factory. This *masse cuite* was of so poor a quality that at the date of writing (October 16) no attempt has been made to separate it.

CARBONATATION.

As pointed out in the experiments in carbonatation, described in Bulletin No. 3, it is evident that the process so long and so successfully practiced with beet juices is also capable of giving good results with cane juices. The process is a very simple one, and consists in adding to the diffusion or expressed juice a large excess of lime and afterwards precipitating the greater part of it with carbonic acid. The whole is then sent to the filter press, where the precipitated carbonate of lime and impurities are separated from the juice.

I had not expected to make a trial of this process, on account of the fact that I feared the appropriation would not be sufficient to carry out both experiments. On arriving here, however, I learned that the sugar company had two filter presses and air-pump and two pans, which could be arranged so as to give the process a trial on a large scale. Accordingly I had a furnace constructed for furnishing carbonic acid, and the rest of the apparatus put into shape for the experiments. The furnace was designed by Mr. G. L. Spencer, who also had a general supervision of the entire work.

Our former experiments had shown that the process had to be carried on somewhat differently from that of the beet juices, owing to the presence in sorghum juice of a large percentage of glucose.

Our first experiments were made with the diffusion juices obtained Thursday, October 8. At that time the effects of the freeze had not shown themselves to any great extent. It was found that about 1½ per cent. of CaO (lime) was sufficient to produce a perfect defecation of the juice. On that day, as nearly as we could estimate it, about 40,000 pounds of juice were carbonated, with the most gratifying results. The juice came from the filter press perfectly limpid and of a delicate amber color. After passing through a sulphur box this juice was sent to the evaporators and reduced to a *masse cuite*, which in color, purity, and

taste was greatly superior to the best product obtained by the usual method. Unfortunately, our improvised air-pump broke down in the middle of the work, which caused the loss of more than half of the diffusion juice obtained, which had already been limed for carbonatation.

The carbonatation of sorghum juice, however, demands the greatest care. If too little lime is added the precipitate does not settle readily and filtration is slow and imperfect. The carbonatation must be continued until all but 0.2 per cent. of the lime has been removed. If more than this remains the juice will darken up and become bitter on boiling. If less than this quantity is left the impurities appear to be redissolved, and a green scum forms on top of the still liquor instead of sinking with the precipitate. With the help of proper test reagents a little experience will enable the operator to carry the carbonatation to a successful completion.

It is found also that the temperature during carbonatation should not be allowed to rise above 40° C. Directly the carbonatation is completed the juice is raised as rapidly as possible to the boiling point and sent at once to the filter press. If allowed to stand the liquor will quickly darken. Foaming is prevented by the addition of a little lard to the sugar and by jets of steam from a perforated pipe near the top of the pan.

Analysis of carbonatated juices.

Number.	Sucrose.	Glucose.	Not sugars.	Total solids.
	Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
1	5.25	1.18	2.25	8.68
2	6.51	1.47	3.00	10.98
3	4.99	0.48	3.72	9.19

The first and second of these represent successful carbonatation. The filter juice was all that could be desired in color and flavor. The *masse cuite* made from it was also of the best quality and has already been noted.

The third analysis represents an unsuccessful carbonatation. Too much lime was left in the liquor, and the *masse cuite* was black and bitter.

In all 100,000 pounds of juice was carbonatated, and I do not hesitate to say that this process of defecation offers every evidence of being the one which should be brought into general use. In large sugar factories the saving in scums alone in one season would pay for the carbonatation plant.

I submit herewith a statement of the analyses made of the juices from the company's mill during the progress of our work.

Analyses of sorghum juices.

Date.	Hour of day.	From first mill. Sucrose.	From first mill. Reduced to sugar.	From first mill. Not sugar.	From first mill. Solids.	From second mill. Sucrose.	From second mill. Reduced to sugar.	From second mill. Not sugar.	From second mill. Total solids.
		Per ct.	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*	*Per ct.*
September 9	....	9.40	....	....	14.75	9.55	.....	... .	14.77
September 11	....	9.72	2.94	2.44	15.30	8.65	3.25	3.00	14.90
September 12	7.30 a. m.	10.48	2.58	3.04	16.10	9.12	2.83	4.35	16.30
September 12	11 a. m.	7.70	3.54	2.36	13.60	8.55	3.20	3.60	15.30
September 12	3 p. m.	9.87	2.20	3.53	15.60	10.59	2.54	2.17	15.30
September 14	10 a. m.	10.73	2.68	2.37	15.78	9.00	2.77	3.83	15.60
September 15	....	11.17	2.10	2.88	16.15	10.51	2.70	3.54	16.75
September 16	....	9.39	3.43	3.68	16.50	8.86	....	.....	15.32
September 17	10.10 a. m.	9.18	3.49	2.10	14.77	8.09	3.62	3.26	14.97
Means	....	9.74	2.87	2.80	15.38	9.21	2.99	2.97	15.47
Coefficient of purity		63.3				59.5			

From the above it is seen that the juice from the second mill is slightly inferior to that from the first. In quantity the two mill juices were about the same.

Annalyses of mixed mill juices.

Date.	No.	Sucrose.	Reduced sugar.	Solids not sugar.	Total solids.
		Per cent.	*Per cent.*	*Per cent.*	*Per cent.*
September 9	1	9.39	....	....	15.67
September 10	2	8.88	....	....	14.77
September 10	3	10.32	....	....	15.30
September 10	4	8.25	3.00	3.15	14.40
September 10	5	9.38	3.24	2.78	15.40
September 14	6	12.25	1.68	2.52	16.45
September 15	7	11.29	2.22	2.80	16.31
September 17	8	7.87	3.91	4.03	15.81
September 21	9	10.49	....	....	16.38
September 25	10	10.13	2.77	2.84	15.74
September 28	11	9.51	3.74	2.94	16.19
September 28	12	10.09	2.71	2.61	15.41
September 29	13	9.68	2.87	3.11	15.66
October 1	14	9.77	2.54	2.59	14.90
October 3	15	10.59	2.85	1.80	15.24
October 5	16	9.88	3.14	3.18	15.20
October 6	17	9.76	2.83	2.54	14.93
October 6	18	9.51	2.80	2.54	14.85
October 7	19	9.34	3.15	2.31	14.80
October 9	20	7.80	3.50	2.21	13.51
October 10	21	7.32	3.50	3.19	14.01
October 10	22	7.69	3.15	3.50	14.34
October 12	23	6.67	3.27	3.56	13.50
October 14	24	5.72	3.96	3.20	12.88
Means		9.23	3.04	2.87	15.07

The above is a fair exposition of the quality of the juices worked by the company during the season. It shows that there is much yet to be done in the culture and improvement of the cane before a product is obtained which is favorable to the production of a large quantity of sugar per ton. I am told by the officers of the company that the season has been unusually unfavorable, a late wet spring and a remarkably early freeze combining to prevent the cane from reaching maturity.

The mean coefficient of purity of the juices worked by the company

is 61.3. I have every reason to believe that by proper culture, fertilizing, and selection, sorghum cane can be produced in which the juices will have a coefficient of purity of 75 to 80. The importance of securing such a cane is even greater than that of extracting all the sugar and properly defecating the juice.

In continuing the experiments in topping cane as soon as the seed heads appear, as described in Bulletin No. 5, the following results were obtained with canes grown as nearly as possible on the same kind of soil and under the same conditions of culture:

Date.	Topped and suckered cane.				Topped canes not suckered.				Cans not touched.			
	Sucrose.	Glucose.	Not sugar.	Total solids.	Sucrose.	Reduced sugar.	Not sugar.	Total solids.	Sucrose.	Reduced sugar.	Not sugar.	Total solids.
	P. ct.	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*	*P. ct.*
September 14	13.22	2.25	2.15	19.62	12.78	2.66	2.26	17.70	13.01	2.12	2.13	17.26
September 15	13.31	2.06	2.56	17.93	12.59	2.22	2.67	17.48	11.35	2.52	2.44	16.31
September 16	12.87	1.65	3.13	17.65	12.90	1.93	2.96	17.29	12.16	2.16	2.27	16.59
September 17	10.37	3.05	2.33	15.75	12.77	1.92	2.76	17.45	14.60	1.29	3.06	18.95
September 18	13.34	1.93	3.13	18.40	12.88	2.66	1.58	17.12	12.25	2.59	1.88	16.72
September 19	12.59	1.98	2.79	17.36	13.00	2.06	3.00	18.06	11.52	2.31	2.63	16.46
September 28	13.49	1.80	3.22	18.51	12.33	2.11	2.97	17.41	12.18	2.06	2.77	17.01
September 29	12.57	1.57	3.14	17.28	11.80	1.76	2.69	16.25	12.74	1.45	3.26	17.45
September 30	11.11	1.84	2.80	15.75	11.42	2.02	3.26	16.70	10.39	1.99	2.60	14.98
September 30	11.67	1.76	2.99	16.44	12.16	1.53	3.48	17.17	11.26	2.08	2.62	15.96
Means	12.45	1.99	2.82	17.26	12.46	2.09	2.76	17.31	12.15	2.06	2.56	16.77
Coefficient	72.1				72.0				72.4			

From the above results it is seen that no appreciable increase of sucrose is obtained by topping and suckering the canes, and therefore it is useless to make further experiments in this direction.

SOLUBLE MATTERS AND WATER.

The total soluble matter in the clean canes, obtained by repeated extractions with water and drying and weighing the exhausted residue, is shown in the following table:

Soluble matter and water in canes.

Number of determination.	Per cent.	Number of determination.	Per cent.
1	88.42	9	89.80
2	88.32	10	89.80
3	90.70	11	91.80
4	90.92	12	91.00
5	90.45	13	91.80
6	90.40		
7	88.80	Average	90.00
8	87.80		

WAHL VACUUM-PAN.

A new kind of evaporator for the continuous reduction of thin to thick liquor in partial vacuo, built by Wahl Brothers, of Chicago, was in operation by the company. The pan worked well and appeared to have little or no inverting effect on the sucrose, as is indicated in the following analyses:

Effect of Wahl pan evaporation.

Thin liquor before passing through pan.				Same after passing through pan.			
Sucrose.	Red sugar.	Solids not sugar.	Total solids.	Sucrose.	Red sugar.	Solids not sugar.	Total solids.
Per cent.	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*	*Per cent.*
10.29	3.53	3.28	17.10	37.44	12.91	8.95	59.30
11.20	4.23	3.44	18.87	40.28	14.91	12.81	68.00
11.18	3.70	4.02	18.90	31.09	10.37	11.76	53.22
10.87	4.51	2.99	18.44	20.54	13.13	2.11	35.78
10.24	2.93	2.58	15.75	26.74	10.32	8 24	45.30
*10.76	*3.74	*3.26	*17.81	*31.22	*12.33	*8.78	*52.32
†60.4				†59.6			

* Means. † Coefficient of purity.

EXPERIMENTS IN AIR EVAPORATION.

Attention was called in Bulletin No. 5 to two forms of apparatus designed to reduce sirup to sugar by means of hot air and at a temperature low enough to prevent any notable inversion of sucrose.

One of these forms of apparatus was designed by Mr. Stuart, of Iowa, and the other by Mr. Denton, of Kansas. Hoping that a trial of these apparatus might result in showing that sugar for general family use might be made in a small way and without expensive apparatus led to a trial of both forms.

This trial was under the immediate supervision of Mr. A. A. Denton.

The results of the experiments were not satisfactory.

The Stuart apparatus gave fairly good results as far as temperature is concerned. The sirup was heated by steam coils in the bottom of a long rectangular tank through which air was forced by devices described in Bulletin No. 5, p. 178.

In the experiments made here the temperature of the body of the sirup was kept at about 180° F. Near the end of the concentration, however, it was impossible to avoid a little scorching of the mass in contact with the pipes.

The apparatus, however, is extremely simple and cheap, and I think with rich cane might be made to yield a fair quantity of sugar.

The apparatus constructed by Mr. Denton consisted of a rectangular box 28 feet high, 4 feet wide, by 2 feet. In this box was a double endless chain carrying two sets of galvanized iron pieces. These pieces dipped into the sirup below and thus carried it towards the top of the shaft.

A blower attached to a heating chamber containing 400 feet of steam pipe threw a current of hot air into the bottom of the apparatus, which ascending through the box came in contact with the thin surface of liquid spread over the galvanized iron surface.

Apparently the heating surface was not sufficiently great, for it was almost impossible to raise the temperature of the liquid within the box to 100° F.

The experiment was therefore abandoned, but Mr. Denton thinks that with a proper heater it will prove successful. The low specific heat of air will render it quite difficult to keep the temperature high enough to allow the escaping air to carry off any considerable quantity of aqueous vapor, but it is possible that some such device will secure the production of sugar on a small scale.

I still, however, adhere to the opinion expressed in Bulletins Nos. 3 and 5, that sugar-making on a small scale is not the rational method of procedure. The experience of the whole world has been that successful sugar-making implies the investment of sufficient capital to secure the best machinery and to work the most economically.

The experiments of the present season have not shown any sufficient reason for a change in that opinion.

DEFECATION WITH ACID ALBUMEN.

A trial was also made with the Wilcox method of purifying sorghum juice with acid albumen.

The method employed was communicated to me by Mr. Wilcox, the inventor.

Following is his description of the process:

"Our first care is that the sorghum juice is from freshly-cut cane, as a few hours makes a great difference in its sugar-yielding qualities, as you are aware.

"The fresh juice being placed in a defecator, and while in the cold state we add 3½ grains of dried egg albumen dissolved in cold water for each pound of juice, and thoroughly incorporate with the same. We next slowly add enough of a solution, consisting of one part of 66° sulphuric acid and seven parts of water, till litmus shows slightly more red than it would if the juice were simply in the natural state.

"The correct acid point is very important, and to determine this we take some of the contents of the defecator in a test-tube and heat it over a lamp to the boiling point and remove. If the liquid is vigorously stirred a few moments a greenish precipitate will gather and a clear liquor remain; if, on the other hand, not enough of the acid solution has been added, the liquor will be cloudy in appearance and contain but a small quantity of precipitate. If such is the case, dip a small splint in the acid solution and add to the contents of the tube till a good precipitate takes place. If too much acid solution has been used in the defecator, no precipitate will form in the tube, nor will the albumen

coagulate, a condition which is very bad, requiring the use of lime at an improper time to correct. This tube test will give the exact state of the contents of the defecator, and by it the correct acid point obtained.

"The heat is now raised till the contents of the defecator stands at a temperature of 190° F., when it is shut off. We now add milk of lime (which will form more precipitate) till the liquor is exactly neutral. Good, sharp, caustic lime should be used, as it will take less of it, and our experience shows that the less we have to use the better. Another point at this stage is to see that the liquors do not grow alkaline after they show neutral, as lime acts somewhat slowly; if such should be the case, add enough of the acid solution till litmus changes to a purplish blue; but the neutral point must be reached to cause a full precipitation, so as not to be bothered with it during concentration.

"After defecation, we pass the juice into a vessel of the same capacity as the defecator, called a separator, and collect the precipitate in the bottom of the cone, from which it is drawn off through the faucet, thereby saving a great deal of labor in straining. If the liquor should still show any cloud, then strain it through a filter-press or bag-filter, when it will be clear and brilliant, of a light lemon color."

Unfortunately we did not have time to make a trial of the process until after the cane had been injured by frost. I am of the opinion that the process carried out as described above will not work any injury to the sucrose in the juice.

The result of the defecation showed that the juice filtered through filter-paper was limpid and of a light lemon color. About 2,000 pounds of it was sent to the filter-press, but in spite of every endeavor it would not pass through. In a few moments the cloths were completely closed, and at the highest pressure obtainable no liquid could pass. The difficulties with the Wilcox method are therefore not chemical but mechanical. I cannot suggest any method by which these mechanical difficulties can be overcome.

In concluding this report, I desire to properly thank Prof. M. A. Scovell and Mr. James Forsythe for the services they rendered in erecting the machinery; and my assistants, Messrs. Clifford Richardson, G. L. Spencer, and John Dugan, for the aid they gave in the laboratory and in the factory.

I desire also to thank Professor Swenson and Mr. W. L. Parkinson, the chemist and manager of the Franklin Sugar Company, for the many courtesies shown us by them, and for the readiness with which they accorded us every facility of manipulation in their power.

GENERAL CONCLUSIONS.

The general results of the experiments may be summarized as follows:

(1) By the process of diffusion 98 per cent. of the sugar in the cane

was extracted, and the yield was fully double that obtained in the ordinary way.

(2) The difficulties to be overcome in the application of diffusion are wholly mechanical. With the apparatus on hand the following changes are necessary in order to be able to work 120 tons per day. (a) The diffusion cells should be made twice as large as they now are; that is, of 130 cubic feet capacity. (b) The opening through which the chips are discharged should be made as nearly as possible of the same area as a horizontal cross-section of the cell. (c) The forced feed of the cutters requires a few minor changes in order to prevent choking. (d) The apparatus for delivering the chips to the cells should be remodeled so as to dispense with the labor of one man.

(3) The process of carbonatation for the purification of the juice is the only method which will give a limpid juice with a minimum of waste and a maximum of purity.

(4) By a proper combination of diffusion and carbonatation the experiments have demonstrated that fully 95 per cent. of the sugar in the cane can be placed on the market either as dry sugar or molasses.

(5) It is highly important that the Department complete the experiments so successfully inaugurated by making the changes in the machinery mentioned above and by the erection of a complete carbonatation outfit.

Respectfully,

H. W. WILEY,
Chemist.